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14. ABSTRACT
The need for high energy density electrical power sources for the soldier could be met with portable fuel cells and on-demand production of hydrogen from ubiquitous logistical fuels like JP-8. Microplasma (plasma contained in sub-mm volumes) reforming is an exciting prospect since it eliminates the drawbacks of catalysts and offers the promise of efficient chemical processing in a compact form. This project seeks to determine the feasibility of microplasma reforming as an effective means of hydrogen production to support compact fuel cells for soldier

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Report Title

Final Report: Reforming of JP-8 in Microplasmas for Compact SOFC Power <500 W (W911NF0710118)

ABSTRACT

The need for high energy density electrical power sources for the soldier could be met with portable fuel cells and on-demand production of hydrogen from ubiquitous logistical fuels like JP-8. Microplasma (plasma contained in sub-mm volumes) reforming is an exciting prospect since it eliminates the drawbacks of catalysts and offers the promise of efficient chemical processing in a compact form. This project seeks to determine the feasibility of microplasma reforming as an effective means of hydrogen production to support compact fuel cells for soldier power. This experimental study exposes various hydrocarbons that are representative of key compounds in JP-8 to microplasma conditions, and evaluates the effectiveness of the extraction of hydrogen from the feed. Detailed experiments permit the determination of conversion of hydrocarbons under various microplasma conditions. Effective models permit understanding of the mechanisms.

Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

Received		Paper
11/29/2012	2.00	P. J. Lindner, R. S. Besser. A Microplasma Reactor for Chemical Process Intensification, Chemical Engineering & Technology, (07 2012): 0. doi: 10.1002/ceat.201100684
11/29/2012	3.00	E.A. Lennon, A.A. Burke, R.S. Besser. Operating modes and power considerations of microhollow cathode discharge devices with elongated trenches, Current Applied Physics, (07 2012): 0. doi: 10.1016/j.cap.2012.01.008
11/29/2012	4.00	R.S. Besser, P.J. Lindner. Microplasma reforming of hydrocarbons for fuel cell power, Journal of Power Sources, (11 2011): 0. doi: 10.1016/j.jpowsour.2010.11.135
11/29/2012	5.00	Peter J. Lindner, R.S. Besser. Hydrogen production by methanol reforming in a non-thermal atmospheric pressure microplasma reactor, International Journal of Hydrogen Energy, (09 2012): 0. doi: 10.1016/j.ijhydene.2012.06.054
11/29/2012	6.00	Besser, R.S., Lindner, P.J.. Microcavity plasma devices, Recent Patents on Engineering, (04 2011): 57. doi:
TOTAL:	**5**	

Number of Papers published in peer-reviewed journals:

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TOTAL:		

Number of Papers published in non peer-reviewed journals:

(c) Presentations

R.S. Besser and P.J. Lindner, "Microplasma Intensification of a Microchemical Reactor," International Congress of Chemical Engineering, International Meeting of AUNQUE, Seville, Spain, June 27, 2012.

R.S. Besser, "Perspectives on Plasma Gasification," DoD Power Sources Focus Group, Army Research Lab, Adelphi, MD, Sept. 28, 2011.

R.S. Besser, P.J. Lindner, E.A. Lennon, S.Y. Hwang, "Hydrogen generation by microplasma reforming of hydrocarbons," Fuel Cell 2010 (Grove Fuel Cell Conference), Zaragoza, Spain, October 6, 2010.

R. S. Besser, "Microplasma Reforming of Hydrocarbons," Naval Research Lab, Washington, DC, July 28, 2010.

R. S. Besser, "Microplasma Reforming of Hydrocarbons," Army Research Lab, Power and Energy Seminar, Adelphi, MD, September 15, 2009.

R.S. Besser, "Reforming of JP-8 Logistical Fuel in a Microplasma for Compact SOFC Power," Topical Symposium on Plasma Processing, AIChE Spring Meeting, Tampa, Florida, April 27, 2009.

R.S. Besser, "Micro and Nanotechnology for Sustainable Energy," Mechanical Engineering Department Seminar, Facultad de Ingeniería Mecánica y Producción, Escuela Superior Politécnica del Litoral (ESPOL), Guayaquil, Ecuador, June 19, 2009; given in the Spanish language.

R.S. Besser, "Production of Hydrogen with Atmospheric Pressure Microplasmas," Department of Chemical Engineering and Materials Science, University of Connecticut, November 17, 2009.

R.S. Besser, "Microplasmas in Microfluidics for Compact Chemical Processing," Graduate Summer Institute on Complex Plasmas, July 30-August 8, 2008, Hoboken, NJ.

R.S. Besser, "Nanotechnology for Alternative Energy,"Amelio Solar, Inc. Energy Seminar, Ewing, NJ, July 21, 2008.

P. Lindner, A. Najem, K. Ciampi, Y. Liu, and R.S. Besser, "Microplasma Reforming for Hydrogen," Research and Entrepreneurship Day, April 30, 2008, Stevens Institute of Technology, Hoboken, NJ.

P. Lindner, A. Najem, W. Zhu, and R.S. Besser, "Microplasma Reforming of JP-8," Workshop on Soldier-Portable Power Systems: Status Review and Research Needs, February 21, 2008, College Park, Maryland.

R.S. Besser, "Microplasma Reforming of JP-8 for Compact SOFC Power (< 500 W)," Army Research Office Advanced Energy Conversion Workshop," Chapel Hill, North Carolina, Sept. 7, 2007.

Number of Presentations: 13.00

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received	Paper

TOTAL:

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received Paper

TOTAL:

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(d) Manuscripts

Received Paper

TOTAL:

Number of Manuscripts:

Books

Received Paper

TOTAL:

Patents Submitted

Patents Awarded

Awards

Invited speaker, Energy Innovation Academic Symposium with Dr. Steven Chu, Oct. 2011.

Henry Morton Distinguished Teaching Professor, Stevens Institute of Technology, 2011-2012. The award is presented to a faculty member "of exceptional teaching ability who has demonstrated a great influence on students in and/or out of the classroom."

Stevens Alumni Association Outstanding Teacher Award, Sept. 7, 2011.

Invited Author, Chemical and Engineering Technology (Wiley), 2011.

Keynote Speaker, Microscale Fuel Processing, Fuel Division, American Chemical Society Annual Meeting, 2007.

Invited member of the Executive Committee, Ninth International Conference on Microreaction Technology (IMRET10), (2007-2008).

Graduate Students

NAME	PERCENT_SUPPORTED	Discipline
Peter Lindner	0.67	
Sang Youp Hwang	0.67	
FTE Equivalent:	**1.34**	
Total Number:	**2**	

Names of Post Doctorates

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Names of Faculty Supported

NAME	PERCENT_SUPPORTED	National Academy Member
Ronald Besser	0.08	
FTE Equivalent:	**0.08**	
Total Number:	**1**	

Names of Under Graduate students supported

NAME	PERCENT_SUPPORTED	Discipline
Anthony Najem	0.05	Chemical Engineering
FTE Equivalent:	**0.05**	
Total Number:	**1**	

Student Metrics

This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period:	1.00
The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:.....	1.00
The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:.....	1.00
Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):	1.00
Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:	0.00
The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense	0.00
The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields:	0.00

Names of Personnel receiving masters degrees

NAME
Sang Youp Hwang
Total Number: 1

Names of personnel receiving PHDs

NAME
Peter Lindner
Total Number: 1

Names of other research staff

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

List of Illustrations and Tables

Statement of the Problem Studied

The development of standalone, compact fuel cell systems to replace batteries for soldier applications critically depends on the emergence of robust reforming approaches for extracting hydrogen from hydrocarbons. For the Army, the ability to effectively process logistical fuels like JP-8 and others into useable feed streams for fuel cells would be of great benefit. Microplasma reforming, that is, processing in plasmas with sub-millimeter geometries, is an emerging approach for compact and efficient chemical processing, and has the potential to enable the transition to a new generation of power sources based on efficient fuel cell technology.

A variety of hydrocarbon reforming approaches have been developed, the most important of which are shown in Table 1[1]. While these processes have met with varying degrees of success, all the approaches listed share one or more of the following limitations: (1) catalysts are required which are subject to the limitations in lifetime imposed by deactivation associated with carbon deposition and/or sulfur sensitivity[2, 3], (2) the temperature requirement is so high that it precludes the use of common materials, results in thermal expansion mismatch problems, and presents significant thermal integration challenges for a compact unit, or (3) the scheme requires further system complexity in the form of additional processing units. For example, pre-reforming may reduce coking and help to reduce sulfur compounds to more easily adsorbed species [4], but it introduces additional processing units to the system, is itself subject to catalytic degradation, requires more energy input, adds cost, decreases reliability, and significantly increases the initial start-up time[5].

Microplasma reforming is attractive because it does not rely on either catalysts or high temperatures for activation[6]. Consequently it does not display sensitivity to catalyst deactivation through poisoning, coking, or coarsening. Since it can be carried out near room temperature, the requirements of the materials of construction of reactors are less stringent. It differs from conventional plasma processing (as found in semiconductor wafer deposition and etching, for example) in that microplasmas have ion and electron densities several orders higher, enabling significantly greater volumetric capacity to break hydrocarbon bonds. Presently, knowledge of the mechanisms controlling the efficiency of microplasma-mediated reactions is sorely lacking; we propose to ameliorate this situation through a basic study of the details of the mechanisms and factors behind these chemical reactions.

A microplasma can be defined as a plasma confined in at least one dimension to 1 mm or less, and is a promising approach to the generation and maintenance of stable, glow discharges at atmospheric pressure [7, 8]. Microplasmas have pd values (p, operating pressure, and d, characteristic plasma dimension) comparable to those of large-volume, low-pressure processing plasmas, but achieve much higher power densities (exceeding 1 kW/cm3) and are generated under conditions that promote the efficient production of reactive radicals and transient molecular species formed via three-body collisions. Pulsed excitation on a sub-microsecond time scale can create microplasmas with significant shifts in both the temperature and energy distribution functions associated with the ions and electrons. This allows for the selective production of chemically reactive species and opens the door to a wide range of new plasma chemical reaction pathways, independent of catalysts. Miniaturization bestows microplasmas with other advantages over conventional plasmas such as higher electron density (up to and even exceeding 1015 cm-3), and a high surface-to-volume ratio which yields extraordinarily low resistances to heat and mass transport. Consequently, plasma microchemical systems have excellent thermal management characteristics and will mix rapidly, producing homogeneous reacting volumes [9]. Despite the strong potential for compact fuel processors, to date we know of no other group investigating this extremely attractive approach to conversion of hydrocarbons to hydrogen.

Due to the robustness of this approach, the overall scheme for a microplasma fuel processor to supply a feed stream to a fuel cell can be simpler than equivalent schemes based on the conventional reforming technologies listed in Table 1. Shown in Figure 1, is a block diagram of a possible implementation example of a solid oxide fuel cell (SOFC)-based power source implementing a microplasma reformer for a DoD application. The fuel is JP-8, a high-energy content logistical fuel consisting of mixed hydrocarbons of several types. The fuel is vaporized and mixed with a secondary gas (air, steam, or other) serving as a diluent or an oxidizer depending on the reaction chemistry to be targeted. The mixture is aspirated into the microplasma reactor which is operating at atmospheric pressure and approximately 50-100°C. The effluent from the reactor is rich in hydrogen, and may also possess some C1 and C2 hydrocarbons, as well as sulfur species largely in the form of H2S. This effluent passes through a ZnO H2S absorber[4], then directly to the SOFC which can process most of these chemical species as fuel or minimally as benign diluents. This basic schematic could be implemented to serve a variety of systems at different

scales and uses including portable (<500 W), vehicular auxiliary (<5000 W), and stationary (>5000 W) applications. The inherent versatility and scalability of the microplasma reforming approach would make these implementations possible.
Another significant benefit of microplasma reforming relates to the environmental compatibility relative to the growing importance of minimizing CO2 emissions. At present there is great pressure to develop new energy sources which in addition to making efficient use of fuel, produce no emissions which could impact the environment or the climate. Every established fuel processing approach results in the generation of CO2 with the associated negative impacts. Moreover, the emission of CO2 in certain DoD applications, such as in Unmanned Undersea Vehicles (UUVs), is undesirable for maintaining stealth operation. Our experimental results indicate that reforming of simple hydrocarbons can be accomplished without appreciable generation of oxides of carbon. Going forward, this suggests that eventual systems based on this approach can be simpler and cheaper than systems which must include measures for CO2 capture in both military and non-military applications.

Summary of Work Completed

We have gained considerable experience with hydrocarbon microplasmas and have significantly increased our knowledge of them as reflected in several presentations and publications [10-15]. In the work to date, we have created functional microplasma reactors and have used them to carry out reaction experiments with hydrocarbon species. Before constructing a reactor, a simple electrostatic model was created (Figure 1) which served as a guide to the design of the initial experimental microplasma reactor chip. These reactors (Figure 2) were fabricated and their operation verified with inert gases (Figure 3). Discerning the instantaneous input of electrical energy into the microplasma reactor is imperative to evaluating the key metrics of energy efficiency, and the creation of an interface of hardware and software which could sample at kilohertz rates and record high voltages (>500 V) and low currents (< 1 mA). We successfully created the functional experimental setup shown in Figure 4.

Hydrocarbon microplasmas were formed using butane and methane (separately), and the current-voltage behavior recorded. We believe that we are the first group to explore butane microplasmas as we find no previous reports in the literature. Chemistry experiments ensued by sampling the microplasma reaction environments with mass spectrometry, and later gas chromatography.

Key results of these experiments are the following: the microplasma environment of butane alone does not produce a net chemical reaction under the conditions we assessed; however, introducing air leads to hydrogen production (Figure 5) which increases with input power (Figure 6). Curiously, the conversion produces no oxides of carbon, and pure nitrogen alone is as effective in causing conversion as air. We conclude that the reaction is a decomposition process rather than an oxidation, and that nitrogen serves to improve plasma reaction efficiency [16].

In order to estimate overall thermal efficiency, we did an energy balance on the microplasma reactor in operation. This involved instrumenting the reactor chip with a downstream thermocouple to assess convective heat transfer and to determine enthalpies of the exiting species. The result of this measurement was an efficiency of 35%, defined as the heating value of the hydrogen produced divided by the combined heating value of butane fuel fed and electrical energy supplied to the plasma. While the efficiency is insufficient for a high-performance compact electrical generation system, it is an encouraging result given the non-optimized system represented by the experiment.

Evidence for conversion by decomposition without generation of CO2 is indicated in Figure 7. The figure shows two mass spectra; before (blue) and after (red) ignition of the butane plasma mixed with diluents. What is remarkable in this case is that the diluent is air. Despite the presence of O2, there is no appreciable formation of oxygenated species, especially oxides of carbon. At the resolution of the measurement, there is no evidence for increase in either the CO2 or the CO signals. Partial pressure values reflect those inside the analyzer, and not inside the reactor; ratios are equivalent in both locations. The hydrogen production is indicated by evolution of the peak at mass = 2 amu; lack of CO2 production is indicated by the unchanged peak height at mass = 44 amu. Nitrogen is responsible for the peak at 28 amu, which shows no change in signal, indicating no CO formation (also 28 amu).

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Technology Transfer

Table 1: Reforming processes used to convert hydrocarbons into hydrogen-rich feed streams for fuel cells [1].

Process	Summary
Partial Oxidation	1200-1500^{o} C process without catalyst or water, difficult to control and downscale; catalytic process more attractive with lower T, lower contact time
Steam Reforming	Catalytic process requiring water, >500^{o} C, endothermic-heat transfer limited
Autothermal Reforming	Catalytic process with water; heat is from partial combustion of fuel, system is simple
Internal Reforming	Process within SOFC anode compartment, on added catalyst or anode
Thermal Cracking of Hydrocarbons	High T process that separates H and C, carbon deposition difficult to control
Pre-reforming	250-500^{o} C catalytic process; cracks high MW hydrocarbons to help reduce coking later

Table 2: Comparison between basic attributes of catalytic and plasma-driven hydrocarbon conversion processes.

Attribute	Catalytic Process	Plasma Process
Sulfur Compounds	Highly Sensitive	Insensitive
Carbon Deposition (Coking)	Highly Sensitive	Insensitive
Reactor Temperature	High	Low
HDS Reactor	Required	Not Required

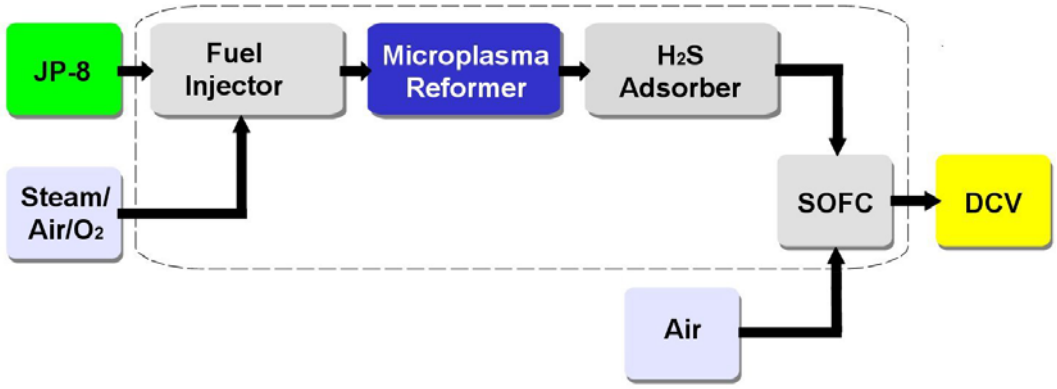

Figure 1: Example of a solid-oxide fuel cell (SOFC) power source having a microplasma-based fuel processor. The hydrocarbon source is high energy content logistical fuel JP-8.

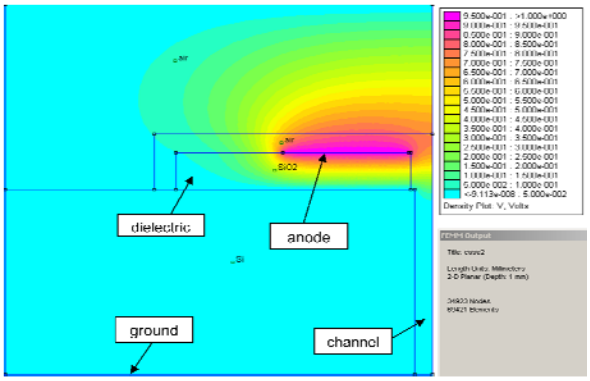

Figure 2: Electrostatic model of microplasma reactor geometry, created to guide design of experimental reactor.

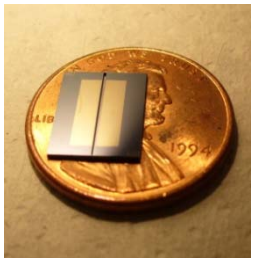

Figure 3: Microplasma reactor chip made by silicon microfabrication.

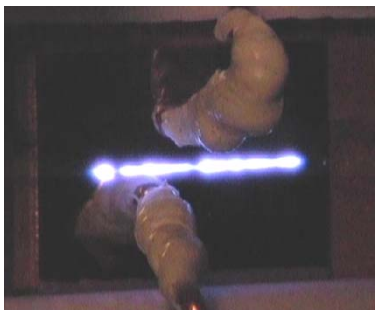

Figure 4: Xenon microplasma formed in reactor chip to characterize plasma operation.

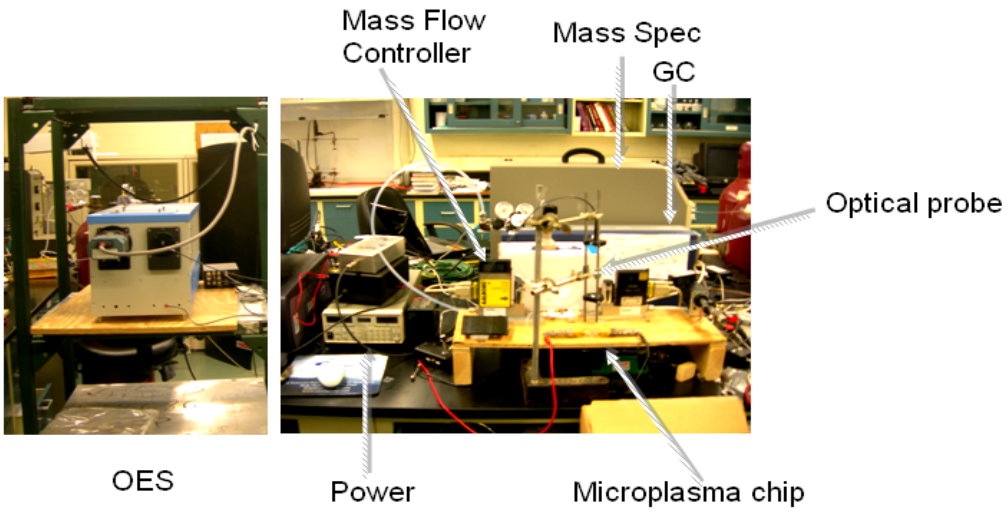

Figure 5: Experimental setup for characterization of microplasma reactor.

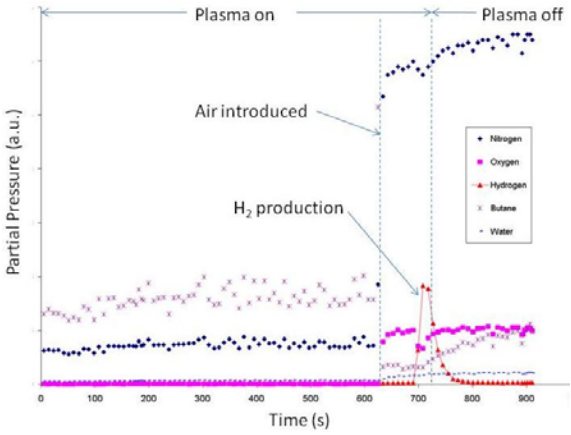

Figure 6: Hydrogen production occurs when air is introduced into the butane plasma.

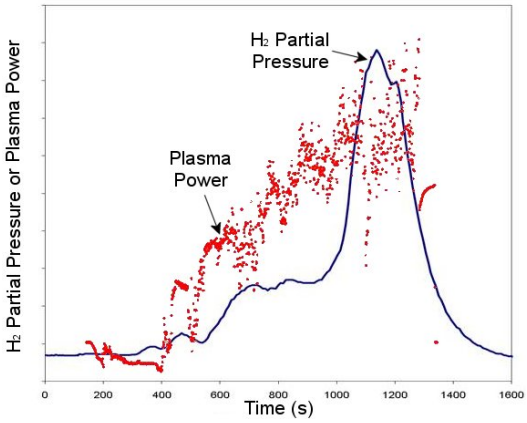

Figure 7: Production of hydrogen from butane in a mixture with air; hydrogen output scales with power. Vertical scale is linear in both partial pressure and plasma power.

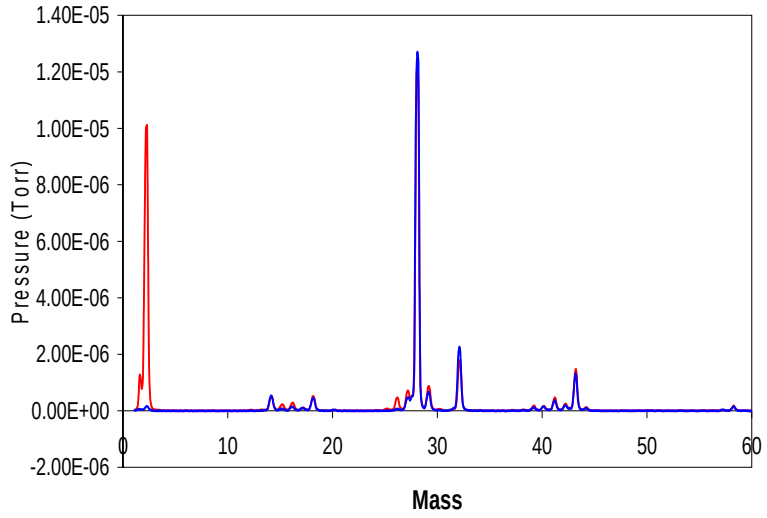

Figure 8: Mass spectra of butane/air mixture (approximately 1:1 ratio by mass) before and after ignition of microplasma [17].